

(19) JAPANESE PATENT OFFICE (JP)  
(12) Official Gazette for Unexamined  
Patent Applications (A)

(11) Japanese Unexamined  
Patent Application No.  
Sho 62-238212

(51) Int. Cl.<sup>5</sup> ID Code(s): Intra-bureau No(s):  
A 61 K 7/021 7306-4C  
7/025 7306-4C

(43) Disclosure Date: October 19, 1987

Request for Examination: No Number of claims: 1 (Total of 9 pages)

(54) Title: Cosmetic Makeup Material

(21) Patent Application No.: Sho 61-82495  
(22) Application Date: April 10, 1986

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Clean Copy of Specification  
(Content Unchanged)

Specification

1. Title of the Invention  
Cosmetic Makeup Material

2. Claim

(1) A cosmetic makeup material comprising an organic silicone resin with the composition shown in Mean General Formula (A), emulsifier, humectant, and water as essential ingredients.

Mean General Formula (A)  $R_n \text{SiO}_{(4-n)/2}$  (where R is a hydrocarbon group with 1 to 6 carbon atoms or a phenyl group, and n is a value from 1.0 to 1.8)

(2) The makeup cosmetic material in Claim 1 in which the organic silicone resin has a three-dimensional structure resulting from the hydrolysis and condensation of organic monochlorosilanes and organic tetrachlorosilane.

(3) The makeup cosmetic material in Claim 1 in which the emulsifier is 1 type or a combination of 2 or more types of substances selected from a group consisting of polyoxyalkylene-substituted organopolysiloxanes, water-absorbing clay minerals, or organically substituted clay minerals.

(4) The makeup cosmetic material in Claim 1 in which the humectant is 1 type or a combination of 2 or more types of substances selected from a group consisting of mucopolysaccharides, acidic mucopolysaccharides, acidic

polysaccharides, neutral polysaccharides, reducing-sugar alcohols, glycols, water soluble proteins, amino acids, amino acid salts and organic acid salts.

(5) The makeup cosmetic material in Claim 1 that comprises a lipstick that contains water.

3. Detailed Description of the Invention  
[Field of Use in Industry]

The present invention concerns a long-lasting and moisture-retaining cosmetic makeup material that is a blend of organic silicone resin having a three-dimensional mesh structure, water, humectant and emulsifier.

[Prior Art]

Cosmetic makeup materials such as foundation, eye shadow, blush, lipstick, mascara, etc normally contain powder, oil, wax, water, etc., and contribute to making the cheeks, eyes, and lips more attractive and beautiful; however, it has been extremely difficult to make the beauty of the finished application last for a long period of time. To make cosmetic makeup longer lasting, methods are generally used such as blending in volatile chain or cyclic silicone oil, or volatile branched hydrocarbons to reduce the ratio of liquid oils that remain after evaporation of the oil base (Japanese unexamined patents Sho 51-151339, Sho 53-142542, Sho 54-28832, Sho 55-20733, Sho 61-40204, etc.), or blending in film-forming ingredients (Japanese granted patent No. 191784 and Japanese unexamined patents 46-32677, Sho 53-94041, etc.) Organic silicone resins are preferred as the film-forming ingredients because they have excellent oil and water

repellency, and they are soluble in chain or cyclic volatile oils and in volatile branched hydrocarbons (Japanese unexamined patents Sho 34-2648, Sho 57-40564, and Sho 61-18708).

However, because these methods involve the use of a solvent, moisture and oils are removed from the surface of the skin, creating a so-called dry, unlubricated condition; after the film is formed, the skin feels as if it is stretched too tight and under pressure, and even after the makeup is removed, various types of irritation (redness, prickly sensation, etc.) associated with the strong adherence of the makeup are observed. In other words, the blending of silicone resin into cosmetic makeup materials has an outstanding effect in terms of rendering the makeup long-lasting, but the skin in places where it is applied (eyes, lips, cheeks, etc.) often becomes dry and irritated.

#### [Statement of the Problem]

The purpose of the present invention is to obtain a cosmetic makeup material for the eyes, lips and cheeks that is safe and has excellent moisture retention while also retaining the long-lasting qualities imparted by organic silicone resins.

#### [Means for Solution of the Problem]

More specifically, the present invention is a cosmetic makeup material comprising an organic silicone resin with a three-dimensional mesh structure and a composition shown in the mean general formula  $R_n \text{SiO}_{(4-n)/2}$  (where R is a hydrocarbon group with 1 to 6 carbon atoms or a phenyl group, and n is a value from 1.0 to 1.8), plus emulsifier, humectant, and water as essential ingredients.

The organic silicone resin used in the present invention should be a suitable combination selected from a group consisting of  $R_3 \text{SiO}_{1/2}$  units,  $R_2 \text{SiO}$  units,  $R \text{SiO}_{2/3}$  units and  $\text{SiO}_2$  units; the ratio of this combination must satisfy the mean general formula  $R_n \text{SiO}_{(4-n)/2}$  (where n is a value from 1.0 to 1.8) and the mean molecular weight of the resin should be ca. 1500-20000.

The aforementioned organic silicone resin is soluble in benzene and is manufactured by several methods. In one method, for example, compounds represented by the general formulas  $R_3 \text{SiX}$ ,  $R_2 \text{SiX}_2$ ,  $R \text{SiX}_3$ , and  $\text{SiX}_4$  (where X represents a group that can undergo hydrolysis, such as chlorine, bromine, fluorine, or an alkoxy group such as a methoxy or ethoxy

group, or an acyloxy group) are crosslinked by a suitable solvent such as toluene, benzene, xylene, etc., depending on the resin composition desired; next a sufficient amount of water is added to obtain the desired hydrolysis and condensation in a suitably acidic solution. The aqueous phase is then removed from this two-phase system, and the remaining resin material is neutralized by a suitable amount of sodium bicarbonate or other alkaline substance; finally, the desired organic silicone resin is obtained by boiling off the solvent.

The organic silicone resin in the present invention comprises 1% to 90% of the total weight of the cosmetic makeup composition.

The organic silicone resin used in the present invention may be used in any of the combinations of units described above, but the most desirable resin is made from a combination of  $R_3 \text{SiO}_{1/2}$  units, which are the most flexible, and  $\text{SiO}_{1/2}$  units which are the most [rigid - poorly legible]. A solvent is essential to dissolve the organic silicone resin and bring out its film-forming properties; chain and cyclic silicone oils and branched hydrocarbon oils can be added to the blend for that purpose, but water and humectant are completely incompatible with these volatile oils, and for that reason, in prior art, water and humectant could not be blended into a cosmetic makeup product that contained silicone resin.

The inventors considered the problems of cosmetic makeup materials containing organic silicone resins, and as a result of their diligent research to add water and humectant to these cosmetic makeup materials, they found that water and humectant could be added with good stability by using 1 type or a combination of 2 or more types of polyoxyalkylene-substituted organopolysiloxanes, water-absorbing clay minerals, or organically substituted clay minerals as an emulsifier, and that the water and humectant had no deleterious effect whatsoever on the film-forming properties of the silicone resin; as a result, the inventors were able to provide a cosmetic makeup material that is safe on the skin and lips both during and after use and that remains very moist. The water and humectant are each added in the amount of 0.1% to 30% with respect to the organic silicone resin, and preferably 2% to 15%. If the amount is less than 0.1%, the moisture-retaining effect is lost, and at the

same time often leads to irritation caused by the strong film-forming properties of the organic silicone resin in the blend. On the other hand, if the amount is greater than 30%, the organic silicone resin becomes chalky and the long-lasting effect of the makeup is lost.

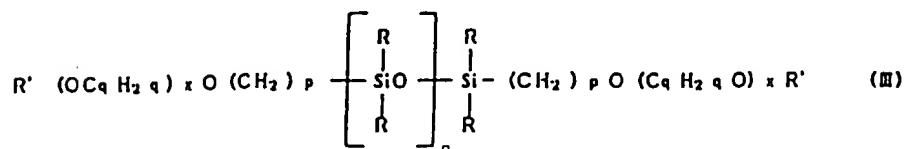
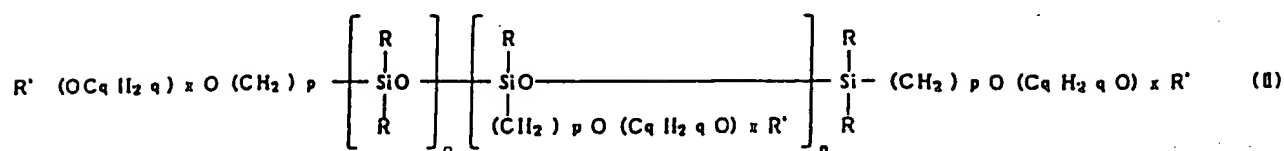
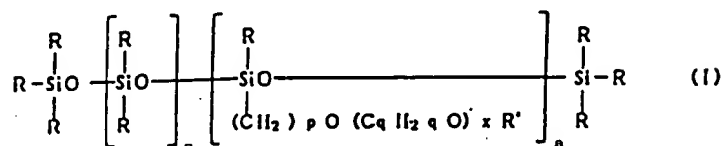
For the humectant, any humectant that is normally used in cosmetics may be used, but it is preferable to use 1 type or a combination of 2 or more types of substances from the group consisting of mucopolysaccharides, acidic mucopolysaccharides, acidic polysaccharides, neutral polysaccharides, reducing-sugar alcohols, glycols, water-soluble proteins, amino acids, amino acid salts and organic salts. Representative compounds include polyethylene glycol, polypropylene glycol, glycerine, 1,3-butylene glycol, xylitol, sorbitol, maltitol, chondroitin sulfuric acid, hyaluronic acid, mucoitin sulfuric acid, charonin sulfuric acid, atherocollagen, sodium lactate, pyrrolidone carboxylic acid salts, bile acid salts, and short-chain soluble collagen.

For the emulsifier, it is preferable to use 1 type or a combination of 2 or more types of polyoxyalkylene-substituted

organopolysiloxanes, water-absorbing clay minerals, or organically substituted clay minerals, and it is most preferable to use a combination of polyoxyalkylene-substituted organopolysiloxanes and water-absorbing clay minerals.

The total amount of emulsifier should be 5% to 200% of the total weight of the water and humectant. General formulas (I), (II), and (III) show polyoxyalkylene-substituted organopolysiloxanes.

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(where in General Formulas (I), (II), and (III),  $R$  is a methyl group or in some cases a phenyl group,  $R'$  is a hydrogen or an alkyl group of 1 to 12 carbons,  $p$  is a number from 1 to 5,  $q$  is a number from 2 to 3, and  $x$ ,  $m$ , and  $n$  are mean values such that the polyoxyalkylene-substituted organopolysiloxane molecule contains 2% to 40% by weight polyoxyalkylene groups and the viscosity of the polyoxyalkylene-substituted organopolysiloxane is 5 to 5000 centistoke at 25°C.)

The water-absorbing clay minerals include natural and synthetic montmorillonite clay minerals such as bentonite, montmorillonite, sauconite, nontronite, saponite, hectorite, vermiculite, and synthetic hectorite (trade name: Laponite).

For the organically-substituted clay minerals, cation-substituted clay minerals such as dioctadecyl dimethylammonium salt-substituted montmorillonite, octadecyl dimethylbenzylammonium salt-substituted montmorillonite, dihexadecyldimethylammonium salt-substituted montmorillonite, etc., and these same clay minerals that have been additionally swollen by nonionic activators may also be used.

Moreover, polyether-substituted dimethyl polysiloxane may be initially blended with the water-absorbing clay minerals or the organically substituted clay minerals and used as a composite. In addition to the aforementioned essential ingredients, the cosmetic makeup material in the present invention may also include waxes, oils, pigments, powders, resins, fragrances, etc., as needed providing the advantage of the present invention is not lost.

#### [Advantage of the Invention]

The cosmetic makeup material in the present invention has excellent long-lasting qualities because of the inclusion of organic silicone resin, emulsifier, water, and humectant., and it has increased moisture-retaining qualities. Below the advantage of the present invention is described using lipstick as an example. The characteristics and formulas for the lipsticks that were prepared are shown in Table-(1) and Table-(2). The various properties of these lipsticks are shown in Table-(3).

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Table-(1) Prepared Lipsticks

Sample No.	Formula Composition
1	Conventional oil-based lipstick
2	Lipstick containing volatile oil
3	Lipstick containing volatile oil and silicone resin
4	Inventive lipstick

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Table-(2) List of Formulas

Sample No.	1	2	3	4
Material				
Liquid paraffin	30	15	10	10
Castor oil	30	15	10	10
dl-2-Heptyl undecanoic acid glycerine	20	14	14	14
Ceresin wax	6	6	6	6
Polyethylene wax	2	2	2	2
Candelilla wax	5	5	5	5
Red iron oxide	0.5	0.5	0.5	0.5
Red #202	1.5	1.5	1.5	1.5
Red #204	1.0	1.0	1.0	1.0
Decamethylcyclopentasiloxane	-	40	25	15
Organic silicone resin *1	-	-	25	25
Polyether-substituted dimethylpolysiloxane *2	-	-	-	1
Synthetic hectorite	-	-	-	3
Purified water	-	-	-	4
Glycerine	-	-	-	2
Total	100.0	100.0	100.0	100.0

- \*1 Organic silicone resin with molecular weight of ca, 3000 and having a mean formula of  $(\text{CH}_3)_{1.35}\text{SiO}_{1.34}$  with a composition of 0.8:1 of  $(\text{CH}_3)_3\text{SiO}_{1/2}$  units: $\text{SiO}_2$  units, .
- \*2 The type shown in General Formula (I). Polyoxyethylene content 15 %, viscosity 220 cs/25°C.
- \*3 Laponite XLG (trade name: made by Laporte, plc, UK)

Table-(3) Properties of Prepared Lipsticks\*

Item	Sample No.	1	2	3	4
Lasting (Utility)		×	△	○	○
Spreading		○	○	○	○
Tightening of skin		○	△	×	○
Moisture of skin after removal of makeup		○	×	×	○
Irritation of skin after removal of makeup		○	△	×	○

\*Evaluation method: Sensory evaluation by 50 female subjects aged 18 to 35.

As shown in Table-(3), Sample 4 (the inventive lipstick) has good lasting properties, and it has about the same qualities as the oil based lipstick in terms of utility and moisture retention during and after use.

#### [Embodiments]

Next, the present invention is described in detail by embodiments, but the present invention is not limited to these embodiments. The quantities of the ingredients are expressed as percent by weight.

The test method used to evaluate both the embodiments and comparison examples was a sensory evaluation performed by 50 female subjects from 18 to 35 years old. A 5-step scale was used for each item; a mean score by the 50 subjects of 4.2 or higher is represented by a circle, a mean score of 4.1 to 3.5 is represented by a triangle inscribed in a circle, a mean score of 3.4 to 2.6 is represented by a triangle, and a mean score of 2.5 or less is represented by an X mark.

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Eye Shadow Stick: Embodiment 1 and Comparison Examples 1 & 2			
	Embodi- ment 1	Compar- ison Example 1	Compar- ison Example 2
Microcrystalline wax	5	5	5
Polyethylene wax	10	10	10
Liquid paraffin	10	10	10
Dimethylpolysiloxane (500 cs)	-	4.6	4.6
Decamethylcyclopentasiloxane	20	20	40
Organic silicone resin *1	20	20	-
Polyether-substituted dimethylpolysiloxane *2	0.5	-	-
Peegam [translit.] HV *3	3	-	-
Purified water	1	-	-
Hyaluronic acid	0.1	-	-
Ultramarine blue	4	4	4
Red iron oxide	1.4	1.4	1.4
Titanium mica	25	25	25
Fragrance	suitable amount	suitable amount	suitable amount
Total	100.0	100.0	100.0

- \*1 Organic silicone resin with molecular weight of ca. 5000 and having a mean formula  $(\text{CH}_3)_{1.23}(\text{C}_6\text{H}_5)_{0.18}\text{SiO}_{1.30}$  with a composition of 0.9:0.1:0.2:1.0  $(\text{CH}_3)_3\text{SiO}_{1/2}$  units: $(\text{C}_6\text{H}_5)_2\text{SiO}$  units: $(\text{C}_6\text{H}_5)\text{SiO}_{2/3}$  units: $\text{SiO}_2$  units,
- \*2 Polyether-substituted dimethylpolysiloxane [type shown in General Formula (II)] (polyoxyethylene content 10 %, viscosity 200 cs/25°C)
- \*3 Trade name of montmorillonite made by R.T. Vanderbilt Company, UK

#### (Method of Preparation)

##### Embodiment 1

Dissolve the hyaluronic acid in purified water and add the polyether-substituted dimethylpolysiloxane and aluminum-magnesium silicate; then add the decamethylcyclopentasiloxane and form an emulsification base. Put the wax, liquid paraffin and organic silicone resin in a container and melt it by heating to 90°C. Add pigment material, fragrance material and the

previously prepared emulsified base to the container, and mix well at 85°C. After removing the air by vacuum, pack the mixture in a designated container, and cool and solidify it to obtain the stick-type eye shadow. During the process some of the decamethylcyclopentasiloxane and purified water will evaporate, and therefore an additional amount is added at the beginning so that the final composition will match that of the recipe.

#### Comparison Examples 1 & 2

Place the ingredients except for the pigment and fragrance materials in a container and melt them by heating to 90°C. After confirming that the ingredients have melted, add the pigment and fragrance materials, and mix well at 85°C. Remove the air by vacuum, and pack the mixture in the designated container to obtain a stick eye shadow. An additional amount of volatile oil is added as a correction in the same manner as in Embodiment 1.

Table-(4) shows the properties of the eye shadows prepared in Embodiment 1, Comparison Example 1 and Comparison Example 2.

Table-(4) Properties of Stick Eye Shadow

	Embodiment 1	Comparison Example 1	Comparison Example 2
Long-lasting	○	○	△
Feeling of tightness	○	×	△
Irritation	○	×	○

In Embodiment 1, the makeup was long lasting, there was no feeling of tightness of the skin, and there was absolutely no prickly feeling or other kind of irritation either during or after use.

#### Embodiment 2, Comparison Example 3 Lipstick

Material	Embodiment 2	Comparison Example 2
Polyethylene wax	7	7
Ceresin wax	4	4
Candelilla wax	8	8
Glyceryl tristearate	18	39
Methylphenylpolysiloxane (1000 cs)	20	40
Dimethylpolysiloxane (1.5 cs)	25	-
Organic silicone resin *1	10	-
Polyether-substituted dimethylpolysiloxane *2	0.7	-
Synthetic hectorite *3	3	-
Purified water	1.5	-
Sodium dl-pyrrolidone carboxylic acid	0.3	-
Sodium lactate	0.5	-
Yellow oxide of iron	1	1
Red oxide of iron	0.3	0.3
Red #204	0.7	0.7
Dibutyl hydroxytoluene	suitable amount	suitable amount
Fragrance	suitable amount	suitable amount
Total	100.0	100.0

\*1 Organic silicone resin with molecular weight of ca. 5000 and having a mean formula of  $(\text{CH}_3)_{1.3}\text{SiO}_{1.1}$  with a composition of 1.5:1 of  $(\text{CH}_3)_3\text{SiO}_{1/2}$  units:  $\text{SiO}_2$  units.

\*2 Polyether-substituted dimethylpolysiloxane [type shown in General Formula (III)].  
Polyoxyethylene-polyoxypropylene content 13%, viscosity 800cs/25°C

\*3 Laponite CP (brand name of Laporte, plc, UK).

#### (Method of Preparation)

In the manner shown in Embodiment 1, mix the purified water, emulsifier, humectant, and methylphenylpolysiloxane uniformly to form the emulsification base. Place the oil and organic silicone resin in a container and heat to 90°C to melt them; add the previously prepared emulsification base, pigment, Dibutyl hydroxytoluene, and fragrance to the container and mix thoroughly at 85°C. Remove the air by vacuum, fill the designated container, and cool and solidify the product to obtain the lipstick. The loading compensation for the volatile oil is performed in the same manner as in Embodiment 1.

#### Comparison Example 3

Place all ingredients except the pigment and fragrance in a container and heat to 90°C to melt them. Add the pigment and fragrance and mix in thoroughly at 85°C. Remove the air by vacuum, fill the designated container, and cool and solidify the product to obtain a lipstick.

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Table-(5) shows the properties of Embodiment 2 and Comparison Example 3

Table-(5) Properties of Lipstick

	Embodiment 2	Comparison Example 3
Long-lasting	○	×
Feeling of tightness	○	○
Irritation (prickly feeling)	○	○
Moistness after removal	○	○

Embodiment 2 was about the same as the oil-based lipstick (Comparison Example 3) in terms of feeling of irritation, but it was much longer lasting,

#### Embodiment 3 Mascara

	Composition (%)
Carnauba wax	7
Paraffin wax	8
Lanolin alcohol	5
Isopar C *1	10
Polyether-substituted dimethylpolysiloxane *2	1
Benton 38 *3	2
Purified water	3
Organic silicone resin *4	30
Octamethylcyclotetrasiloxane	20
Chondroitin sulfate	0.3
Atherocollagen	0.7
Iron oxide (black)	7.5
Talc	5.5
Total	100.0

\*1 Branched isoparaffin made by Esso Corporation.

\*2 Polyether-substituted dimethylpolysiloxane [type shown in General Formula (I)]. Polyoxyethylene content 25%, viscosity 400 cs/25°C

\*3 Dioctadecyldimethylammonium salt-substituted montmorillonite (trade name: Benton 38, NL Chemicals, USA)

\*4 Dow Corning QF1-3593A

#### (Method of Preparation)

Dissolve the chondroitin sulfate and atherocollagen in water, and after mixing in the polyoxy-substituted dimethylpolysiloxane and Benton 38, add the Isopar C and disperse uniformly to form the emulsification base. Place the wax and lanolin alcohol in a container and melt them at 90°C, then add the emulsification base and the powdered ingredients, and mix thoroughly. Remove the air by vacuum and place in a designated container with a lid to obtain mascara.

The mascara thus obtained was very long-lasting, and no irritation, etc., was felt even after the mascara was removed.

#### Embodiment 4 Solid Foundation

	Composition (%)
Microcrystalline wax	3
White petrolatum	5
decamethylcyclopentasiloxane	25
Organic silicone resin *1	25
Polyether-substituted dimethylpolysiloxane *2	1
Kunipia C *3	1
Purified water	2
1,3-butylene glycol	0.5
Glycerine	0.5
Iron II oxide	20
Iron Oxide	3
Kaolin	14
Fragrance	Suitable amount
Total	100.0

\*1 Organic silicone resin with molecular weight of ca. 8000 and having a mean formula of  $(\text{CH}_3)_{0.31}(\text{C}_6\text{H}_5)_{0.85}\text{SiO}_{1.43}$  with a composition of 5.67:1 of  $(\text{C}_6\text{H}_5)\text{SiO}_{3/2}$  units:  $(\text{CH}_3)_2\text{SiO}$  units.

\*2 Polyether-substituted dimethylpolysiloxane [type shown in General Formula (I)]. Polyoxyethylene content 20%, viscosity 300 cs/25°C

\*3 Trade name of montmorillonite [translit.] made by Kunisaki Koka, K.K. [poorly legible]

## (Method of Manufacture)

Dissolve the 1,3-butylene glycol and glycerine in water, mix in the Kunipia C and polyether-substituted dimethylpolysiloxane with a roller to form the emulsification base. Place the wax, white petrolatum, decamethylcyclopentasiloxane and the organic silicone resin in a container and melt to uniformity at 90°C. Add the emulsification base, pigment, and fragrance and mix thoroughly at 85°C. Remove the air by vacuum and fill a designated, pot-shaped container at 70°C with the mixture to obtain a solid foundation.

The solid foundation thus obtained was extremely long-lasting and the kind of caking observed over time with conventional foundations did not occur. Moreover, there was no feeling of tightness of the skin during use, and after use the skin was so moist that the subjects could not believe this was a kind of product that forms a film.

## Embodiment 5 Lip Coating

	Composition (%)
Octamethylcyclotetrasiloxane	40
Methylphenylpolysiloxane	2.49
Organic silicone resin *1	50
Laponite XLC *2	2
Glycerine	0.3
Propylene glycol	0.2
Purified water	5
Red #202	0.01
Total	100.0

\*1 The organic silicone resin used in Embodiment 1

\*2 A synthetic hectorite made by Laporte, plc, UK.

## (Method of Preparation)

Disperse the methylphenylpolysiloxane, humectant and the water in the Laponite XLG to form the emulsification base. Dissolve the silicone resin in the octamethylcyclotetrasiloxane; add this solution and the Red #202 to the emulsification base and stir gently. Pour into a bottle with a lid to obtain a lip coating for applying over lipstick.

The lip coating thus obtained was applied over lipstick and greatly improved the lasting power of the lipstick underneath. Both during and after use, the subjects did not feel that they were wearing a lip coating.

## Embodiment 6 Pressed Blush

	Composition (%)
Purified water	1
Benton 27 *1	0.3
Maltitol	0.3
Organic silicone resin *2	15
Decamethylcyclopentasiloxane	15
Mica	30
Talc	30
Titanium-coated mica	5
Red #202	0.2
Titanium dioxide	1.2
Lanolin	2
Fragrance	suitable amount
Total	100.0

1\* Brand name of NL Chemicals:

dimethylbenzyl octadecyl ammonium salt-substituted montmorillonite

2\* Silicone wax KR114 made by Shinetsu Kagaku, K. K.

## (Method of Preparation)

Add the maltitol to the purified water, and add these to make a uniform dispersion with the Benton 27, organic silicone resin, and decamethylcyclopentasiloxane. After mixing the powdered ingredients, lanolin and fragrance in a Henschel mixer, crush them in a pulverizer. Add the pulverized material to the emulsification base and mix thoroughly. Press-mold into the designated shallow container, and allow the product to harden and set in an air-tight container to obtain the blush.

The blush thus obtained was very long-lasting; although it was applied to the most sensitive part of the face, it did not feel tight on the skin and even after it was removed the skin continued to feel moist.

Applicant: Shiseido Company, Ltd.



Amendment (Self-Initiated)

May 16, 1986

To the Hon. Commissioner of the Japanese Patent Office,  
Michiro Azanuki

1. Title of Item  
Patent Application: Sho 61-82495

2. Title of Invention  
Cosmetic Makeup Material

3. Person Making Amendment

Relationship to Item: Applicant  
Address: 7-5-5 Ginza, Chuo-ku, Tokyo  
Name: Shiseido Company, Ltd.  
Agent: (name illegible--covered by official seals)

4. Amendment to:  
Entire Specification

5. Details of Amendment  
To provide a clean copy of the specification as described on  
attached sheet. (No change in content)

End